



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 141d



This Standard Reference Material (SRM) is intended primarily for use in validating microchemical procedures for the determination of carbon, hydrogen, and nitrogen in organic matter. SRM 141d is supplied in crystalline form in a 2 g unit.

Certified Value and Uncertainty: The purity of this material was assessed at NIST by differential scanning calorimetry (DSC), gas chromatography (GC), and direct probe mass spectrometry (DPMS). Based upon these measurements, the certified purity and expanded uncertainty of the acetanilide are given below.

Certified Purity as Mass Fraction (in %): 99.92 ± 0.01 [1]

The uncertainty in the certified value is expressed as an expanded uncertainty, U , at the 95 % level of confidence, and is calculated according to the method described in the ISO Guide [3]. The expanded uncertainty is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the measurement error. The coverage factor, k , is determined from the Student's t -distribution corresponding to the appropriate associated degrees of freedom and 95 % level of confidence.

Theoretical Chemical Composition: Pure acetanilide has the following theoretical chemical composition expressed as fractions (in %) of its total relative molecular mass [2].

Element	Fractions (in %)
Carbon	71.089
Hydrogen	6.711
Nitrogen	10.363
Oxygen	11.837

NIST recommends that the theoretical chemical composition be used for this material. The analytical techniques used to generate the supplemental information validated the theoretical composition of this material within their experimental error.

Expiration of Certification: The certification of this SRM lot is valid until **30 September 2007**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, the certification is invalid if the SRM is contaminated or modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by J.C. Colbert.

Gaithersburg, MD 20899
Certificate Issue Date: 10 April 1998

Thomas E. Gills, Chief
Standard Reference Materials Program

Analytical measurements at NIST were performed by R.M. Parris, M.P. Urquiza, L.K. Walton, and M.J. Welch of the NIST Analytical Chemistry Division.

The overall direction and coordination of the technical activities were under the chairmanship of M.J. Welch of the NIST Analytical Chemistry Division.

Statistical analysis was provided by L.M. Gill of the NIST Statistical Engineering Division.

NOTICE AND WARNING TO USERS

Storage: The SRM should be stored in its original bottle at temperatures between approximately 20 °C and 25 °C. It must be tightly re-capped after use and protected from excessive moisture and light.

Instructions for Use: SRM 141d is not hygroscopic under ordinary conditions of storage as described above, and can be used without preliminary drying.

SOURCE AND ANALYSIS

Source of Material: The SRM material was obtained from Fluka Chemical Corporation, Ronkonkoma, NY.

Analytical Approach: Analyses for purity were performed by NIST. Ten samples, selected by a stratified random process across the filling run, were analyzed by DSC. All exhibited high purity (>99.9 %) and no trend with respect to filling order was found. A small unidentified impurity was found by DPMS, but its level could not be estimated. Two samples were analyzed by GC-FID and found to have small impurity levels consistent with the DSC data. No measurable impurities were found by GC/MS.

SUPPLEMENTAL INFORMATION

To confirm the theoretical chemical composition for carbon, hydrogen, and nitrogen, three collaborating laboratories performed the analyses. Fifty bottles representative of the entire bottled lot were selected by a stratified random sampling. Each laboratory tested three taken from the group of fifty, chosen such that one bottle came from the first third of the bottling run, one from the second third, and one from the final third. Analyses were performed in duplicate from each of the three bottles for each of the three elements: carbon, hydrogen, and nitrogen. The three laboratories were: Atlantic Microlab, Inc., Norcross, GA; Galbraith Laboratories, Inc., Knoxville, TN; and Schwartzkopf Microanalytical Laboratory, Inc., Woodside, NY. The data from the three laboratories fell within the ranges presented in Table 1.

Table 1. Range of Values (Fractions) for Measured^a Relative Atomic Masses for Carbon, Hydrogen, and Nitrogen in SRM 141d

Element	Range Fraction (in %)
Carbon	71.01 – 71.25
Hydrogen	6.65 – 6.80
Nitrogen	10.22 – 10.49
Oxygen ^b	11.59 – 11.93

^a These results are provided to demonstrate user experience with this material.

^b Oxygen content is calculated by assuming that all that is not carbon, hydrogen, or nitrogen must be oxygen [$\% \text{ O} = 100 - (\% \text{ C} + \% \text{ H} + \% \text{ N})$].

REFERENCES

- [1] Taylor, B.N., "Guide for the Use of the International System of Units (SI)" NIST Special Publication 811, 1995 Ed., (April 1995).
- [2] IUPAC Commission on Atomic Weights and Isotopic Abundances, Atomic Weights of the Elements 1995," Pure & Appl. Chem., **68**, p. 2339, (1996).
- [3] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed. ISO, Geneva, Switzerland, (1993): see also Taylor, B.N. and Kuyatt, C.E., "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," NIST Technical Note 1297, U.S. Government Printing Office, Washington DC, (1994).

It is the responsibility of users of this SRM to assure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: Phone (301) 975-6776 (select "Certificates"), Fax (301) 926-4751, e-mail srminfo@nist.gov, or via the Internet <http://ts.nist.gov/srm>.